
पानी आधारित लेखन स्याही के लिए रंग —
विशिष्टि
(पहला पुनरीक्षण)

Dyes for Water-Based Writing Inks —
Specification
(First Revision)

ICS 87.080

© BIS 2023



भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुर शाह ज़फर मार्ग, नई दिल्ली - 110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI - 110002
www.bis.gov.in www.standardsbis.in

July 2023

Price Group 8

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Printing Inks, Stationery and Allied Products Sectional Committee had been approved by the Chemical Division Council.

The quality of writing ink will, among other factors, depend upon the quality of the dye used in the ink. The manufacturers of inks had been experiencing considerable difficulty in obtaining the requisite quality of dye from indigenous sources. It was, therefore, considered important to formulate a standard for dyes used in ink industry. It was expected that this standard will help the producers of ink to procure the dyes of assured quality in the country. The dyes covered under this standard include ink blue, acid green, acid violet, nigrosine, direct black, crocein scarlet, eosine, acid fast scarlet, tartrazine yellow and acid black.

This standard was first published in 1977. In this revision, all amendments, Reference clause and Packing and Marking clause have been incorporated in the standard.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard***DYES FOR WATER-BASED WRITING INKS — SPECIFICATION***(First Revision)***1 SCOPE**

This standard prescribes the requirements and the methods of sampling and test for dyes used in the manufacture of water-based writing inks.

2 REFERENCES

The standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

<i>IS No.</i>	<i>Title</i>
IS 265 : 2021	Hydrochloric acid — Specification (<i>fifth revision</i>)
IS 266 : 1993	Sulphuric acid — Specification (<i>third revision</i>)
IS 323 : 2009	Rectified spirit for industrial use — Specification (<i>second revision</i>)
IS 364 : 1993	Fanlight catch — Specification
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 2263 : 1979	Methods of preparation of indicator solutions (<i>first revision</i>)
IS 4395 : 1987	Glossary of terms relating to inks and allied industry (<i>first revision</i>)
IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 4395, in addition to the following shall apply.

3.1.1 Approved/Standard Sample

The sample accepted by the purchaser or inspection authority as the basis of supply or manufacture.

4 REQUIREMENTS**4.1 Description**

The dyes shall have hue number, colour index number, chemical name, molecular mass and equivalent mass as given in Table 1.

4.2 Identification

The material shall comply with the identification requirements given in Table 2 when tested by the methods prescribed in Annex A reference to the relevant clauses of Annex A is given in col (13) of the table.

4.3 The material shall also comply with the requirements laid down in Table 3 when tested by the methods prescribed in Annex A reference to the relevant clauses of Annex A is given in col (13) of the table.

4.4 Presence of Starch, Dextrin and Gum

The material shall pass the test prescribed in **A-14**.

5 PACKING AND MARKING**5.1 Packing**

Unless otherwise agreed between the purchaser and the supplier the material shall be packed in sound, clean and dry tinplate containers fitted with lever lids.

5.2 Marking

The containers shall be legibly marked with the following information:

- Description of the material;
- Tare and net mass;
- Manufacturer's name and/or recognized trade-mark, if any; and
- Batch number in code or otherwise to enable the lot and date of manufacture to be traced from records.

5.2.1 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the

Bureau of Indian Standards Act, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

of the material, number of tests to be performed and the criteria for judging the conformity of the material to the requirements of this specification shall be as prescribed in Annex B.

6 SAMPLING

6.1 The method of drawing representative samples

Table 1 Description of Dyes for Water-Based Writing Inks
(Clause 4.1)

SI No.	Name of the Dye	Hue No.	Colour Index No.	Chemical Name	Molecular Mass	Equivalent Mass
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Ink blue	Acid Blue 93	42780	Ammonium salt of triphenyl triamino triphenylcarbinol trisulphonic acid	—	—
ii)	Acid green	Acid green 16	444025	4-4' dimethylamino-phenylmethane naphthyl-3.6' disodium sulphonate	684 (as acid)	—
iii)	Acid violet	Acid Violet 17	42650	N N-ethyl benzoyl aniline formaldehyde, N N diethyl aniline	740 (as acid)	385
iv)	Nigrosine	Acid black 2	50420	Sodium salt of sulphonated nigrosine base which is a complex mixture of phenylated indulines of unspecified structure	—	—
v)	Direct black	Direct black	30235	Sodium salt of benzene azo-3 6 disulfo-8-amino-1-naphthol-7-azo-diphenylazom-phenylene diamine	787.74 (as acid)	61.47
vi)	Crocein scarlet M00	Acid red 73	27290	Sodium salt of benzene azobenzene azo-2-naphthol-6 8 disulfonic acid	512.4 (as acid)	64.06
vii)	Eosine	Acid red 87	45380	Disodium salt of tetrabromofluoreseein	691.9	—
viii)	Acid fast scarlet	Acid red 18	16255	Trisodium salt of 1-(4-sulfo-1-naphtylazo)-2-hydroxy naphthalene 6 8 disulfonicacid	604.5	151.1
ix)	Tartrazine yellow	Acid yellow 23	19140	Trisodium salt of 4-(4'-sulfol-phenylazo)-l-(4'-bulphophenyl)-5-hydroxy pyrazol-3- carboxylic acid	534.4	133.6
x)	Acid black	Acid black 1	20470	Sodium salt of 1-nitrobenzene azo-3 6-disulfo-l-amine-8-naphthol azo benzene	572	40.89

Table 2 Identification Requirements of Dyes for Water-Based Writing Inks
(Clause 4.2 and A-2.2.1, A-3.2.1)

SI No.	Characteristic	Identification Requirements										Method of Test, Ref to
		Ink blue	Acid Green	Acid Violet	Nigrosine	Direct Black	Crocein Scarlet M00	Eosine	Acid Fast Scarlet	Tratrazine Yellow	Acid Black	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
i)	Physical appearance	Lumps, granules or powder with deep brown golden lustre	Brownish black powder	Brownish violet powder with lustre	—	Greyish black power	Scarlet power	Scarlet powder	Dark scarlet powder	Orange powder	Dark brown powder	—
ii)	Colour of aqueous solution (0.5 percent) <i>mlv</i>	Blue	Bluish green	Violet		Greenish Black	Red	Bluish red with a yellowish green fluorescence	Deep Red	Yellow	Black	—
iii)	Action of acid											
a)	Dilute hydrochloric acid	Blue solution	Yellowish brown solution	Dark green Solution	—	Blackish solution with precipitate	Blackish brown solution with precipitate	Orange solution with precipitate	Deep Red solution	Yellow solution	Black solution with precipitate	A-2
b)	Concentrated hydrochloric acid	Blue solution	Brownish orange solution	Reddish brown solution	—	Dull reddish black solution with precipitate	Blackish brown solution with precipitate	Orange solution with precipitate	Deep Red solution	Yellow solution	Bluish black solution with precipitate	

Table 2 (Concluded)

Sl No.	Characteristic	Identification Requirements										Method of Test, Ref to
		Ink blue	Acid Green	Acid Violet	Nigrosine	Direct Black	Crocein Scarlet M00	Eosine	Acid Fast Scarlet	Tratrazine Yellow	Acid Black	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
c)	Dilute sulphuric acid	Blue solution	Yellowish brown solution	Violet solution	—	Reddish black solution with precipitate	Reddish brown solution with precipitate	Orange solution with precipitate	Reddish orange solution	Yellow solution	Greenish black solution with precipitate	A-3
d)	Concentrated sulphuric acid	Reddish Brown	Yellowish brown solution	Reddish yellow solution	—	Bluish black solution with precipitate	Reddish violet solution with precipitate	Yellow solution	Violet solution	Yellow solution	Deep green solution with precipitate	
iv)	Action of Alkali											
a)	Sodium hydroxide solution	Reddish brown solution	Bluish green solution	Bluish violet	—	Greyish blue solution with precipitate	Reddish brown solution with precipitate	Reddish	Reddish brown solution	Reddish yellow solution	Greenish solution	
b)	Sodium carbonate Solution	Violet solution	Bluish green solution	Bluish violet Solution	—	Bluish grey solution	Red solution	Red solution	Deep red solution	Yellow solution	Bluish black solution	

Table 3 Requirements of Dyes for Water-Based Writing Inks
(Clause 4.3 and B-5.1.1, B-5.1.2)

Sl No.	Characteristic	Requirements for										Method of Test, Ref to
		Ink blue	Acid Green	Acid Violet	Nigrosine	Direct Black	Crocein Scarlet M00	Eosine	Acid Fast Scarlet	Tratrazine Yellow	Acid Black	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
i)	Dye content, percent by mass	see Note 1	see Note 1	see Note 1	see Note 1	57 ± 2	62 ± 2	—	80 ± 2	80 ± 2	62 ± 2	A-4
ii)	Strength, percent											
a)	By spectrophotometer or colorimeter	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	100 ± 2	—	A-5
b)	By dyeing	Close match with the approved standard sample										A-6
iii)	Volatile matter, percent by mass <i>Max</i>	10.0	5.0	5.0	—	10.0	5.0	10.0	5.0	10.0	10.0	A-7
iv)	pH value (of aqueous solution)	4.0 to 4.5	8.0 ± 0.5	8.0 ± 0.5	—	10.0 ± 0.5	10.0 ± 0.5	10.0 ± 0.5	8.0 ± 0.5	8.0 ± 0.5	10.0 ± 0.5	A-8
v)	Matter Insoluble in water percent by mass, <i>Max</i>	0.2	1.0	1.0	—	1.0	1.0	1.0	1.0	1.0	1.0	A-9

Table 3 (Concluded)

Sl No.	Characteristic	Requirements for										Method of Test, Ref to
		Ink blue	Acid Green	Acid Violet	Nigrosine	Direct Black	Crocein Scarlet M00	Eosine	Acid Fast Scarlet	Tratrazine Yellow	Acid Black	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)
vi)	Colour value (of 0 002 5 percent aqueous, solution)	0.9R+ 2.0B	4.0Y + 0.3R + 9.0B	13.5R + 7.1B	—	0.7Y + 3.5R + 3.0B	2.0Y + 7.7R	10.8Y + 23.9R (see Note 2)	0.8Y + 7R (see Note 2)	23Y + 3.7R + 0.3B	4.0Y + 2.9R + 3.0B	A-10
vii)	Shade				Close match with the approved/standard sample							A-11
viii)	Calcium oxide (as Cao), percent by mass, <i>Max</i>	0.8	—	—	6.0	—	—	—	—	—	—	A-12
ix)	Sulphated ash percent by mass, <i>Max</i>	5.0	—	—	—	—	—	—	—	—	—	A-13
NOTES												
1 The determination of strength by reduction with titanium trichloride is not possible as these dyes do not belong to azo group of dyes.												
2 The values are for 0.5 percent solution.												

ANNEX A
(Clauses 4.2 and 4.3, Tables 2 and 3)

METHODS OF TEST FOR DYES FOR WATER-BASED WRITING INKS

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be used in tests.

NOTE — 'Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 TEST FOR ACTION OF ACID

A-2.1 Reagents

A-2.1.1 *Dilute Hydrochloric Acid* — 10 percent (v/v)

A-2.1.2 *Concentrated Hydrochloric Acid* — See IS 265

A-2.1.3 *Dilute Sulphuric Acid* — 10 percent (v/v)

A-2.1.4 *Concentrated Sulphuric Acid* — See IS 266

A-2.2 Procedure

Take 0.5 g of the dye and dissolve in 100 ml of water. Transfer 5 ml of this solution to a test tube and add 5 ml of dilute hydrochloric acid. Observe the colour of the solution and the precipitate, if any carry out similar tests with concentrated hydrochloric acid, dilute sulphuric acid and concentrated sulphuric acid.

A-2.2.1 The material shall satisfy the requirement of the test if the action produced is the same as specified in Table 2.

A-3 TEST FOR ACTION OF ALKALI

A-3.1 Reagents

A-3.1.1 *Sodium Hydroxide Solution* — 10 percent (m/v)

A-3.1.2 *Sodium Carbonate Solution* — 10 percent (m/v)

A-3.2 Procedure

Take 0.5 g of the dye and dissolve it in 100 ml of water. Transfer 5 ml of this solution to a test tube and add 5 ml of sodium hydroxide solution observe the colour of the solution and the precipitate, if any. Carry out similar tests with sodium carbonate solution.

A-3.2.1 The material shall satisfy the requirement of the test if the action produced is the same as specified in Table 2.

A-4 DETERMINATION OF DYE CONTENT

A-4.1 Reagents

A-4.1.1 *Ammonium Thiocyanate Solution* — 20 percent (m/v)

A-4.1.2 *Dilute Hydrochloric Acid* — 25 percent (m/v)

A-4.1.3 *Titanium Trichloride Solution*

Approximately 0.025 N, prepared as follows. Prepare a 15 percent (m/v) solution of titanium trichloride. Take 30 ml of the solution. Filter it through a thick pad of glass wool. Add this solution to a previously boiled mixture of 1 litre of water and 60 ml of concentrated hydrochloric acid. Mix this solution by passing into it a current of inert gas, such as carbon dioxide or nitrogen, for about 15 min. Store the mixture in a bottle in an atmosphere of carbon dioxide.

NOTE — Carbon dioxide may be supplied by Kipp's apparatus. The solution shall be stored in a bottle painted black to protect it from light.

A-4.1.4 *Ferric Ammonium Sulphate Solution* — Saturated solution (see IS 2263).

A-4.2 Procedure

A-4.2.1 Weigh accurately about 1 g of the dye and dissolve it in hot water. Pour the solution into a 500 ml graduated flask and make the volume with water to 500 ml mark. Mix well.

A-4.2.2 Pipette out 50 ml of the solution into a 500 ml reduction flask containing 50 ml of dilute hydrochloric acid and 50 ml water which have been previously brought to boil. Start passing carbon dioxide into the solution and simultaneously heat the contents to boil.

A-4.2.3 Pass carbon dioxide in the storage bottle containing titanium trichloride solution and immediately draw 50 ml of titanium trichloride solution by a pipette in which carbon dioxide has been passed previously and add this solution to the dye

solution (see A-4.2.2). Immediately boil the contents for 10 min to 15 min for complete reduction. Cool the flask in an ice-bath. Maintain continuous flow of carbon dioxide throughout the test. Add 10 ml of ammonium thiocyanate indicator solution. Titrate the mixture against ferric ammonium sulphate solution to a permanent change of colour. Note the volume of ferric ammonium sulphate solution (see Note).

NOTE — The volume of ferric ammonium sulphates solution required for titration should not be less than 10 ml.

A-4.2.4 Carry out a blank by following the same procedure and the same quantities of the reagents but using 50 ml of water in place of the dye solution.

A-4.3 Calculation

$$\text{Dye content, percent by mass} = \frac{(B - A) \times N \times D \times V}{H \times C \times M \times 10}$$

where

B = volume in ml of ferric ammonium sulphate solution required for the blank (see A- 4.2.4);

A = volume in ml of ferric ammonium sulphate solution required for the dye;

N = normality of ferric ammonium sulphate solution;

D = molecular mass of the dye;

V = volume of dye solution (see A-4.2.1);

H = number of hydrogen atoms consumed per molecule of the dye;

C = volume in ml of aliquot taken for titration (see A-4.2.2); and

M = mass in g, of the sample taken for the test.

A-4.4 Carry out the test for approved/standard sample also under identical conditions using standard dye in place of sample dye.

NOTE — Each azo group requires four nitro group, six hydrogen group and two atoms of hydrogen for reduction.

A-5 DETERMINATION OF STRENGTH BY SPECTROPHOTOMETER OR COLORIMETER

A-5.1 By Spectrophotometer Method

A-5.1.1 Reagents

A-5.1.1.1 Buffer solution — of approximate pH 5 for

ink blue, acid green, acid violet nigrosine (as acid black), crocein scarlet and eosine (as acid red), and 9 for direct black.

A-5.1.2 Procedure

A-5.1.2.1 Weigh accurately 1.0 g of the dye and dissolve it in hot water. Pour the solution into 1 litre graduated flask and make up the volume with water up to the mark. Mix well.

A-5.1.2.2 Pipette out 10 ml aliquot from the dye solution and pour it in 1 litre graduated flask. Add 50 ml of the buffer solution and finally make up the volume with water to 1 litre mark. Mix well.

A-5.1.2.3 Prepare solution of the approved/standard samples as described in A-5.1.2.1 and A-5.1.2.2.

A-5.1.2.4 Prepare two absorption 1 cm cells for use which are satisfactorily matched to each other in light transmittance when filled with water. In one cell, take water as a reference solution and in another cell, take the dye solution of the sample as prepared in A-5.1.2.2. Measure the optical density of the solution at the following wavelengths

Ink blue	550 nm
Acid green	605 nm
Acid violet	530 nm
Nigrosine as acid black	618 nm
Direct black	484 nm
Crocein scarlet	510 nm
Eosine as acid red	510 nm

A-5.1.2.5 Similarly take the optical density of the approved/standard dye solution prepared in A-5.1.2.3 following the same procedure as in A-5.1.2.4.

A-5.1.3 Calculation

Calculate the percentage strength of the dye sample considering the optical density of the approved/standard sample as 100 percent

$$\text{Strength of dye, percent} = \frac{D_1}{D_2} \times 100$$

where

D_1 = optical density of the sample; and

D_2 = optical density of the approved/standard dye.

A-5.2 By Photoelectric Colorimeter

A-5.2.1 Reagent

A-5.2.1.1 Buffer solution — same as A-5.1.1.1

A-5.2.2 Procedure

Weigh accurately 1.000 g of the sample and dissolve it in hot water. Pour the solution into 1 litre volumetric flask and make up the volume with water up to the mark and mix well. Pipette out 10 ml aliquot of the solution and transfer it to another 1 litre graduated flask. Add 50 ml of the buffer solution and finally make up the volume with water up to 1 litre mark. Mix well. Prepare the solution of the approved/standard dye in a similar manner. Measure the percentage transmission of the sample solution on a photoelectric calorimeter using suitable filters. Similarly measure the percentage transmission of the approved/standard dye solution.

A-5.2.3 Calculations

Calculate the percentage strength of the dye considering the strength of the approved/standard solution as 100 percent

$$\text{Strength of the dye, percent} = \frac{T_1}{T_2} \times 100$$

where

T_1 = percentage transmission of the sample; and

T_2 = percentage transmission of the approved/standard dye.

A-6 DETERMINATION OF STRENGTH BY DYEING

A-6.1 Reagents

A-6.1.1 Sodium Sulphate (Calcined) Solution — 10 percent (m/v)

A-6.1.2 Sodium Chloride Solution — 10 percent (m/v)

A-6.1.3 Formic Acid — 10 percent (m/v)

A-6.1.4 Soda Ash Solution — 10 percent (m/v)

A-6.2 For Acid Dyes

A-6.2.1 Preparation of Hanks for Dyeing

A-6.2.1.1 Hanks of scoured 100 percent pure woollen knitting yarn (except for direct black in which case cotton yarn shall be used, (see A-6.3) having

no finishing chemicals, blueing or fluorescent brightening agents, shall be used. Each hank shall weigh $5 \text{ g} \pm 0.1 \text{ g}$.

A-6.2.1.2 Pretreatment of hanks

The hank shall be heated in warm water (60°C) for 10 min, squeezed evenly to contain approximately its own mass of water, cooled and entered into the dye-bath.

A-6.2.2 Preparation of Dyestuff Solution

A-6.2.2.1 Weigh accurately 1.0 g of the dye paste it thoroughly with cold water. Add hot water and dissolve the dye. If necessary, heat the solution till it becomes clear. Dilute the solution to 1 litre with cold water.

A-6.2.2.2 Similarly prepare solution of the approved/standard dye following the procedure mentioned in A-6.2.2.1.

A-6.2.2.3 Depth of shade

Use medium shades (about 1.0 percent) as they show variation in strength more clearly than dark shades.

A-6.2.3 Dyeing Procedure

Pipette out 50 ml of the dye sample solution prepared as in A-6.2.2.1. Similarly pipette out separately 50 ml the approved/standard dye solution in other dye vessel. Add 5 ml of sodium sulphate solution, 1 ml of formic acid and 250 ml of water in each dye vessel. Stir the dye liquor. Place them in water-bath and raise the temperature to 50°C . Introduce wetted hanks in each dye vessel and turn them frequently so as to obtain level dyeing. Work for 10 min at this temperature remove the hanks from the dye vessels, add 1 ml of formic acid in each vessel and stir well. Introduce the hanks into the dye-bath and turn the hanks frequently slowly raise the temperature of dye-bath to boiling within 20 min continue the dyeing at boil for 45 min. At the end of dyeing squeeze the dyed hank and rinse with warm water (60°C). Dry the hanks in an oven or in a drier at temperature not exceeding 70°C .

A-6.3 For Direct Black

A-6.3.1 Preparation of Hanks for Dyeing

A-6.3.1.1 A sufficient number of hanks of scoured, bleached, unmercerized cotton yarn having no finishing chemicals, blueing or fluorescent

brightening agents, shall be used in this test. Each hank shall weigh 10 g $\pm$ 0.1 g (see Notes below).

NOTES

1 Any yarn normally used in the laboratories for carrying out trials or yarn of the following requirements is suitable for this test.

- a) Count — 2/40 s or 2/60 s;
- b) Twist per metre — 750; and
- c) Cuprummum fluidity — not more than 5.

2 If the mass of hank is not 10 g $\pm$ 0.1 g then it should be weighed accurately and the amount of dyes and the chemicals to be taken should be calculated accordingly.

A-6.3.1.2 Pretreatment of test hanks

The hank shall be treated in boiling water for 10 min, squeezed evenly to contain approximately its own mass of water, cooled and entered into the dye-bath.

A-6.3.2 Preparation of Dyestuff Solutions

A-6.3.2.1 Weigh accurately 10 g of the sample. Paste it thoroughly with cold water. Add hot water and dissolve the dye if necessary, heat the solution till it becomes clear. Dilute the solution to 1 litre with cold water

A-6.3.2.2 Similarly, prepare solution of the standard dye by following the procedure mentioned in **A-6.3.2.1** but taking the standard dye instead of the sample dyes.

A-6.3.3 Dyeing Procedure

Pipette out separately 100 ml of solution of the dye sample prepared as in

A-6.3.2.1 In the dye vessel so as to get one percent depth (see **A-6.2.2.3**). Similarly, pipette out separately 100 ml of the solution of approved/standard dye in the other dye vessel. Add 190 ml of water and 10 ml of sodium chloride solution or sodium sulphates solution in each dye vessel. Stir the dye liquors and enter the wetted hanks at 40 °C. Turn the hanks frequently so as to obtain level dyeings. Slowly raise the temperature of dye-bath to 80 °C within 20 min. Remove the hanks from the dye-bath, add second lot of 10 ml of sodium chloride solution or sodium sulphate solution in each dye vessel and stir well. Enter the hanks into the dye-bath and raise the temperature of the dye-bath to boil and continue the dyeing for 40 min. At the end of dyeing, squeeze the dyed hanks returning the squeezed

out solution back to respective dye-baths and rinse them well in cold water dry the hanks in oven at temperature not exceeding 70 °C.

A-6.4 The material shall be taken to have satisfied the requirement of the test, if the colour of the wool/cotton when dyed with the material is not different from the colour of the wool/cotton dyed with the approved/standard dyestuff.

A-7 DETERMINATION OF VOLATILE MATTER

A-7.1 Procedure

Accurately weigh 2 g to 3 g of the sample in a dried ground-glass petri dish with cover (about 5 cm diameter and 3 cm height). Uncover the dish and expose the material for three h in an oven at 105 °C. Remove the dish, replace the cover, cool it in a desiccator and weigh. Repeat the process till constant mass is obtained.

A-7.2 Calculation

$$\text{Volatile matter, percent by mass} = \frac{M}{M_1} \times 100$$

where

M = loss in g, of the material; and

M_1 = mass in g, of the material taken for the test.

A-8 DETERMINATION OF pH VALUE

A-8.1 Procedure

Accurately weigh 0.5 g of the dye and dissolve it in 100 ml of hot water. Cool it to room temperature and determine the pH value by means of a suitable pH meter.

A-9 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-9.1 Procedure

Dissolve with stirring about 10 g of the accurately weighed material in about 500 ml of cold water. Filter the solution through a weighed Gooch crucible and wash the residue repeatedly with cold water till the filtrate is colourless. Dry the Gooch crucible in an oven at 105 °C for 3 h. Cool it in a desiccator and weigh. Repeat the process till constant mass is obtained.

A-9.2 Calculation

Matter insoluble in water, percent by mass

$$= \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g, of the crucible and the residue;
and

M_2 = mass in g, of the material taken for the test.

A-10 DETERMINATION OF COLOUR VALUE OF AQUEOUS SOLUTION (0.0025 PERCENT SOLUTION)**A-10.1 Procedure**

Weigh accurately 0.250 g of the dye and dissolve it in hot water. Transfer the solution into 1 litre graduated flask, giving the necessary water washings to the container till final water washing is free from the colour. Make up the volume with water to 1 litre mark. Shake it well.

A-10.2 Pipette out 10 ml aliquot of the solution and transfer it quantitatively into 100 ml graduated flask as described in **A-10.1**. Make up the volume with water up to 1 litre mark. Shake it well. Take this prepared solution in a 1 cm cell and take the readings on Lovibond Tintometer in terms of yellow (Y), red (R), and blue (B).

A-11 TEST FOR SHADE (ON 0.5 PERCENT AQUEOUS SOLUTION)**A-11.1 Procedure**

A-11.1.1 Weigh accurately 1.250 g of the dye. Dissolve it in hot water. Transfer the solution into 250 ml graduated flask giving necessary water washings to the container till final washing is free from the colour. Make up the volume to 250 ml with water and shake it well.

A-11.1.2 Transfer the solution into 500 ml beaker. Take the shade of the solution by just dipping a Whatman filter paper piece (6 cm × 10 cm) into the solution. Dry the paper by hanging it at room temperature for about an h.

A-11.1.3 Similarly, prepare the solution of the approved/standard dye as in **A-11.1.1** and take the shade of the standard solution as in **A-11.1.2**.

A-11.2 The material shall be taken to have satisfied the requirement of the test if the shade of the dyed piece of the sample solution matches with the shade of the dyed piece of the standard solution.

NOTE — The shade test papers (as prepared in **A-11.1.2** and **A-11.1.3**) may further be examined by reflectance photometer at different wavelengths or by Lovibond Tintometer and may be compared.

A-12 DETERMINATION OF CALCIUM OXIDE (CaO)

A-12.1 General — Two methods are prescribed, namely, (a) volumetric method, and (b) EDTA method.

A-12.1.1 Volumetric Method**A-12.1.1.1 Reagents**

A-12.1.1.1.1 Dilute hydrochloric acid — 120 (v/v) and 12 (v/v)

A-12.1.1.2 Ammonium chloride — solid

A-12.1.1.3 Ammonium nitrate — solid

A-12.1.1.4 Concentrated nitric acid — see IS 264

A-12.1.1.5 Dilute ammonium hydroxide — 1 : 1 (v/v)

A-12.1.1.6 Ammonium hydroxide ammonium nitrate wash solution

Add 5 ml of ammonium hydroxide solution (r. d 0.88) and 2 g of ammonium nitrate to 1 litre of water.

A-12.1.1.7 Ammonium oxalate solution — saturated

A-12.1.1.8 Dilute sulphuric acid — 1 : 1 (v/v)

A-12.1.1.9 Standard potassium permanganate solution — 0.05 N

A-12.1.1.10 Methyl red indicator — Dissolve 0.1 g of methyl red in 100 ml of rectified spirit (see IS 323).

A-12.1.2 Procedure

A-12.1.2.1 Weigh accurately 1 g to 2 g of the dye, dried over sulphuric acid in a desiccator, in a platinum dish or crucible Heat over a Bunsen burner raising the temperature slowly and keeping it red hot till the dye is completely ashed. Cool the crucible and transfer the contents to a beaker digest with 20 ml of hydrochloric acid (1 : 2), dilute with 50 ml of water and boil. Filter and wash the residue with dilute hot hydrochloric acid

(1 : 20). Determine calcium in the filtrate.

A-12.1.2.2 Take the solution in a 400 ml beaker, dilute to 200 ml, add 3 g of solid ammonium chloride and boil with 1 ml of concentrated nitric acid. Cool and neutralize with dilute ammonium hydroxide using 3 to 4 drops of methyl red indicator and then a slight excess of ammonia. Keep the solution on the water-bath for 10 min to 12 min; filter through a Whatman No. 40 or 4 litre filter paper and wash with hot ammonium hydroxide-ammonium nitrate wash solution till the filtrate is free from chlorides.

A-12.1.2.3 Concentrate the filtrate to 200 ml and make the solution ammoniacal. Bring to boil and add 10 ml to 15 ml of saturated ammonium oxalate solution. Continue boiling for 10 min and then place the beaker on a water-bath for about 2 h, keeping the solution distinctly ammoniacal. Filter off the precipitate of calcium oxalate through a filter paper and wash with water at room temperature till the washings are free from oxalate.

A-12.1.2.4 Transfer the precipitate to the beaker in which calcium was precipitated by punching the filter paper, wash down with 15 ml of dilute sulphuric acid and finally with 5 to 6 times with hot water through a jet to bring the calcium oxalate into solution. Dilute the contents of the beaker to about 200 ml. Heat to 60 °C and titrate the solution while hot with standard potassium permanganate solution until a stable pink colour is obtained.

A-12.1.3 Calculation

Calcium oxide, percent by mass = $\frac{A \times B}{C} \times 2.803$
where

A = volume in ml of standard permanganate solution consumed;

B = normality of the standard permanganate solution; and

C = mass in g, of the sample taken.

A-12-2 EDTA Method

A-12.2.1 Reagents

A-12.2.1.1 Dilute hydrochloric acid — 4 N approximately

A-12.2.1.2 Dilute ammonium hydroxide — 15 percent (m/m)

A-12.2.1.3 Methyl red indicator solution — Dissolve 0.15 g in 500 ml of water.

A-12.2.1.4 Ammonium chloride ammonium hydroxide buffer solution (pH 10)

Mix 350 ml of ammonium hydroxide (20 percent, m/m) with 54 g of ammonium chloride. Dilute with water and make up the volume to 1 000 ml.

A-12.2.1.5 Eriochrome black T indicator — Dissolve 0.1 g of eriochrome black T in 25 ml of methyl alcohol.

A-12.2.1.6 Standard calcium solution

0.01 M. Weigh exactly 0.500 4 g of calcium carbonate and dissolve in minimum quantity of dilute hydrochloric acid make up the volume with water to one litre in a volumetric flask.

A-12.2.1.7 Standard ethylene diamine tetraacetate (EDTA) solution

Dissolve 3.72 g of disodium ethylene diamine tetraacetate dihydrate in water and make up the volume to 1 litre and standardize as follows:

Take 10 ml of standard calcium solution in a conical flask. Add 20 ml of water, 1 ml of eriochrome black T indicator and 2.5 ml of ammonium hydroxide-ammonium chloride buffer solution. Heat to 40 °C to 50 °C and then titrate with EDTA solution, maintaining the temperature between 40 °C and 50 °C until the colour changes to distinct blue.

$$\text{Molarity of EDTA solution} = 10 \times \frac{M_1}{V_1}$$

where

M_1 = molarity of standard calcium solution; and

V_1 = volume in ml of EDTA solution used for titration.

A-12.2.2 Procedure

Weigh accurately 1 g of the dye dried over sulphuric acid in a desiccator in a silica dish and heat it over the flame of a bunsen burner and then finally ignite in an electric furnace for 1 h at about 500 °C to 600 °C dissolve the ash in 10 ml to 15 ml of dilute hydrochloric acid (do not boil), and neutralize with dilute ammonium hydroxide using methyl red indicator Heat over a water-bath for 10 min to 15 min

to digest any precipitate formed. Filter the solution to remove any iron, aluminium, etc. Wash the residue with hot water to remove calcium chloride completely.

A-12.2.2.1 Treat the filtrate and the washings with about 5 ml of ammonium hydroxide-ammonium chloride buffer solution and titrate with EDTA solution using eriochrome black T as indicator till blue end point is obtained.

A-12.2.2.2 Calculation

Calculate the calcium oxide (CaO) content, percent by mass, on the basis that 1 ml of 0.01 molar EDTA solution is equivalent to 0.000 56 g of calcium oxide (CaO).

A-13 DETERMINATION OF SULPHATED ASH

A-13.1 Procedure

Ignite a 7 cm silica basin in a muffle furnace, cool in a desiccator and weigh place about 2 g of the dye in the crucible and weigh accurately. Heat the crucible on a low flame until the material is completely carbonized. Burn off the carbonaceous matter by further heating in a muffle furnace. When the ash is practically white in colour, cool the crucible, add 5 drops of concentrated sulphuric acid (*see* IS 266) and heat to fumes on a burner. When all the sulphuric acid has volatilized, cool the crucible in a desiccator and weigh accurately.

A-13.2 Calculation

Sulphates ash, percent by mass = $\frac{100 M}{M_1}$
where

M = mass in g, of the ignited ash; and
 M_1 = mass in g, of the material taken for the test.

A-14 TEST FOR PRESENCE OF STARCH, DEXTRIN AND GUM

A-14.1 Principle

Rectified spirit precipitates both starch and dextrin from aqueous solution of dyes while rectified spirit precipitates gum, starch and dextrin

from acidic solution of dye.

A-14.1.1 Reagents

A-14.1.1.1 Rectified spirit — 95 percent (*see* IS 323)

A-14.1.2 Concentrated Hydrochloric Acid — *See* IS 265

A-14.1.3 Phloroglucinol — powder

A-14.1.4 Aniline

A-14.1.5 Iodine Solution — approximately 0.1 N

A-14.2 Procedure

A-14.2.1 Detection of Gum, Starch and Dextrin

Treat 10 ml of 2 percent dye solution with 20 ml of rectified spirit and after thorough shaking, dilute the solution with an equal volume of water. Allow the solution undisturbed for 2 h. Formation of any precipitate or gelatinous matter indicates the presence of starchy materials and gums.

A-14.2.2 Distinction between Gum and Dextrin

Acidify the dye solution with concentrated hydrochloric acid and add enough rectified spirit. Filter the precipitate, if any, wash with rectified spirit and treat with concentrated hydrochloric acid. Divide the liquid into two parts:

- To one part add phloroglucinol. A rose violet colour indicates the presence of gum; and
- To second part, boil and add aniline. A rose red colour again shows the presence of gum. Negative reactions to above two tests show the absence of gum but presence of dextrin.

A-14.2.3 Distinction Between Dextrin and Starch

Filter the solution containing the precipitate obtained in **A-14.2.1**. Take the precipitate in water, boil until the entire precipitate dissolves. To this solution add few drops of iodine solution. Brown colouration indicates dextrin, whereas blue colouration indicates starch.

ANNEX B
(Clause 6.1)

SAMPLING OF DYES FOR WATKR-BASED WRITING INKS

B-1 GENERAL

B-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1.1 Samples shall be taken in a protected place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 Samples shall be placed in clean, dry, airtight glass or other suitable containers on which the material has no action.

B-1.5 Each sample container after filling shall be sealed airtight with a stopper, and marked with full

particulars of the material (*see* 4.2).

B-2 SCALE OF SAMPLING**B-2.1 Lot**

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot for ascertaining the conformity of the material to the requirements of the specification.

B-2.2 The number (n) of containers to be chosen from the lot shall depend on the size of the lot and shall be in accordance with Table 4.

Table 4 Number of Containers to be Selected for Sampling
(Clause B-2.2)

Sl No.	Lot Size	No. of Containers to be Chosen
(1)	(N)	(n)
	(2)	(3)
i)	Up to 50	2
ii)	51 to 100	3
iii)	101 to 150	4
iv)	151 to 300	5
v)	301 to 500	6
vi)	501 to 800	7
vii)	801 to 1 300	8
viii)	1 301 and above	9

B-2.3 These containers shall be chosen at random from the lot and an order to ensure the randomness of selection, procedure given in IS 4905 may be followed.

B-3 TEST SAMPLES AND REFEREE SAMPLE**B-3.1 Preparation of Test Samples**

B-3.1.1 Draw with an appropriate sampling instrument a small portion of material from different parts of each of the containers selected. The total quantity of the

material so drawn from each container shall be sufficient for triplicate determination of all the characteristics given under 4.

B-3.1.2 Thoroughly mix all portions of material drawn from the same container. Out of these portions a small but equal quantity shall be taken from each selected container and shall be mixed up well together so as to form a composite sample. The composite sample shall be divided into three equal parts, one for the purchaser another for the supplier and the third to be used as the referee sample.

B-3.1.3 The remaining portions of the material from each container shall be divided into three equal parts. These parts shall be transferred immediately to thoroughly dried bottles which shall then be sealed airtight with stoppers and labelled with all particulars of sampling given in 5.2. The material in each such sealed bottle shall constitute test sample. These individual samples shall be separated into three identical sets of test samples in such a way that each set has a test sample representing each container selected. One of these three sets shall be sent to the purchaser another to the supplier and third shall be used as referee sample.

B-3.2 Referee Sample

The referee sample consisting of a composite sample and a set of test sample marked for this purpose shall bear the seals of the purchaser and the supplier to be used in case of a dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for dye content, strength and colour value, shall be conducted individually on each of the samples constituting a set of test samples.

B-4.2 Tests for the remaining characteristics given in 3 shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 Individual Samples

Test results for the dye content, strength and colour

value shall be recorded.

B-5.1.1 Dye Content and Strength

The mean and range of test results for each of these characteristics shall be calculated as given below:

$$\text{Mean } (\bar{X}) = \frac{\text{The sum of test results}}{\text{Number of tests}} \text{ and}$$

Mean (R) = The difference between the maximum and the minimum values of the test results.

The lot shall be considered to have satisfied and the requirements for the relevant characteristics if the expression $X \pm 0.6 R$ lies within corresponding limits given in Table 3.

B-5.1.2 Colour Value

The lot shall be considered to have met the requirements of colour value if all the test results on each of the individual samples satisfy the corresponding requirements given in Table 3.

B-5.2 Composite Sample

For the remaining characteristics prescribed under 4, the test results on the composite sample shall meet the corresponding requirements given in 4.1, 4.2 and 4.3.

B-5.3 The lot shall be declared as conforming to the requirements of this specification if B-5.1 and B-5.2 are satisfied, otherwise not.

ANNEX C
(Foreword)
COMMITTEE COMPOSITION

Printing Inks, Stationery and Allied Products Sectional Committee, CHD 14

<i>Organization</i>	<i>Representative(s)</i>
Govt Printing, Govt of West Bengal, Kolkata	SHRI SUBIR KUMAR MANDAL (Chairperson)
All India Federation of Master Printers, New Delhi	SHRI HARJINDER SINGH
All India Printing Ink Manufacturers Association Limited, Mumbai	SHRI RAVINDRA GANDHI SHRI R SRIDHARAN (<i>Alternate</i>)
All India Print-tech Professionals Forum, Kolkata	SHRI PARTHA PRATIM SANYAL
Consumer Voice, New Delhi	SHRI M. A. U. KHAN SHRI K. C. CHAUDHARY (<i>Alternate</i>)
DIC India Limited, Noida	SHRI U. CHOWDHURI SHRI SAROJ PANDA (<i>Alternate</i>)
Directorate of Printing, New Delhi	SHRI D. K. JAIN SHRI K. K. PURI (<i>Alternate</i>)
Flint Group, Noida	SHRI SANJEEV BANSAL SHRI DINESH AHUJA (<i>Alternate</i>)
Government of Indian Stationery Office, Kolkata	SHRI BISHAMBER DHAR (<i>Alternate</i>)
Hubergroup India Pvt Ltd, Mumbai	SHRI RAJEEV SHARMA SHRI RAJEEV RANJAN (<i>Alternate</i>)
Indian Institute of Packaging, New Delhi	DR TANWEER ALAM SHRI SUBODH KAMALAKAR JUIKAR (<i>Alternate</i>)
Kokuyo Camlin Limited, Mumbai	SHRI MANIK J. SALUNKHE SHRIMATI SAYALI SURAJ SARFARE (<i>Alternate</i>)
Kumarappa Handmade Paper, Jaipur	DR SAAKSHY AGARWAL
National Archives of India, New Delhi	SHRI RAM SWAROOP DR SUTAPA CHAKRAVARTY
National Test House, Ghaziabad	SHRI BUDDH PRAKASH
Ordnance Factory Ambajhari, Nagpur	SHRI RAHUL KOMALKAR
Sakata Inx (India) Limited, New Delhi	SHRI VIJAY SHANKAR GUPTA SHRI SUNIL K. CHHABRA (<i>Alternate</i>)
Security Printing and Minting Corporation India Limited, Delhi (SPMCIL)	SHRI S. MAHAPATRA DR D. K. RATH (<i>Alternate</i>)
Shriram Institute for Industrial Research, Delhi	DR MANMOHAN KUMAR DR VINAY TYAGI (<i>Alternate</i>)

<i>Organization</i>	<i>Representative(s)</i>
SICPA India Limited, New Delhi	SHRI ARUNVIG SHRI A. B. SACHAN (<i>Alternate</i>)
Siegwerk Inks, Bhiwadi	SHRI JATIN TAKKAR DR JOERG PETER LANGHAMMER (<i>Alternate</i>)
The Regional Institute of Printing Technology, Kolkata	SHRI SHANKHYA DEBNATH SHRI KRISHNENDU HALDER (<i>Alternate</i>)
Times Group, New Delhi	SHRI SNEHASIS ROY SHRI ANUP KUMAR PAL (<i>Alternate</i>)
Toyo Ink India Private Limited, Noida	SHRI K. S. MURTHY SHRI VIVEK RASTOGI (<i>Alternate</i>)
Whale Stationery Products Limited, Delhi	SHRI MUKESH GUPTA SHRI ASEEM GUPTA (<i>Alternate</i>)
Western Printing Group, Survey of India, Delhi	SHRI EQUERAR AHMAD
Yansefu Inks and Coating Pvt Ltd, Gurugram	SHRI JITENDRA SHARMA SHRI NEELAKAMAL MOHAPATRA (<i>Alternate</i>)
BIS Directorate General	SHRI A. K. LAL, SCIENTIST 'F'/SENIOR DIRECTOR AND HEAD (CHEMICAL) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary
SHRI SAGAR SINGH
SCIENTIST 'D'/JOINT DIRECTOR
(CHEMICAL), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act, 2016* to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Copyright

BIS has the copyright of all its publications. No part of these publications may be reproduced in any form without the prior permission in writing of BIS. This does not preclude the free use, in the course of implementing the standard, of necessary details, such as symbols and sizes, type or grade designations. Enquiries relating to copyright be addressed to the Head (Publication & Sales), BIS.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website- www.bis.gov.in or www.standardsbis.in

This Indian Standard has been developed from Doc No.: CHD 14 (20168).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones: 2323 0131, 2323 3375, 2323 9402

Website: www.bis.gov.in

Regional Offices:

	Telephones
Central : 601/A, Konnectus Tower -1, 6 th Floor, DMRC Building, Bhavbhuti Marg, New Delhi 110002	{ 2323 7617
Eastern : 8 th Floor, Plot No 7/7 & 7/8, CP Block, Sector V, Salt Lake, Kolkata, West Bengal 700091	{ 2367 0012 2320 9474
Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, Chandigarh 160019	{ 265 9930
Southern : C.I.T. Campus, IV Cross Road, Taramani, Chennai 600113	{ 2254 1442 2254 1216
Western : Plot No. E-9, Road No.-8, MIDC, Andheri (East), Mumbai 400093	{ 2821 8093

Branches : AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. CHANDIGARH. CHENNAI. COIMBATORE. DEHRADUN. DELHI. FARIDABAD. GHAZIABAD. GUWAHATI. HIMACHAL PRADESH. HUBLI. HYDERABAD. JAIPUR. JAMMU & KASHMIR. JAMSHEDPUR. KOCHI. KOLKATA. LUCKNOW. MADURAI. MUMBAI. NAGPUR. NOIDA. PANIPAT. PATNA. PUNE. RAIPUR. RAJKOT. SURAT. VISAKHAPATNAM.